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ORIGINAL PAPER

Effects of two modification methods on the mechanical properties of wood flour/recycled plastic blends composites: addition of thermoplastic elastomer SEBS-g-MAH and in-situ grafting MAH

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Abstract: The effect of maleic anhydride grafted styrene-ethylene- butylene-styrene block copolymer (SEBS-g-MAH) and in-situ grafting MAH on mechanical, dynamic mechanical properties of wood flour/recycled plastic blends composites was investigated. Recycled plastic polypropylene (PP), high-density polyethylene (HDPE) and polystyrene (PS), were mixed with wood flour in a high speed blender and then extruded by a twin/single screw tandem extruder system to form wood flour/recycled plastic blends composites. Results show that the impact properties of the composites were improved more significantly by using SEBS-g-MAH compatibilizer than by using the mixtures of MAH and DCP via reactive blending in situ. However, contrary results were observed on the tensile and flexural properties of the corresponding composites. In General, the mechanical properties of composites made from recycled plastic blends were inferior to those made from virgin plastic blends, especially in elongation break. The morphological study verified that the interfacial adhesion or the compatibility of plastic blends with wood flour was improved by adding SEBS-g-MAH or in-situ grafting MAH. A better interfacial bonding between PP, HDPE, PS and wood flour was obtained by in-situ grafting MAH than the addition of SEBS-g-MAH. In-situ grafting MAH can be considered as a potential way of increasing the interfacial compatibility between plastic blends and wood flour. The storage modulus and damping factor of composites were also characterized through dynamic mechanical analysis (DMA).

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Introduction

With the rapid development of plastic industry, large amounts of plastic wastes have been generated all over the world, especially in China. It is becoming a troublesome problem to dispose of these plastic wastes properly. Several methods such as landfills, incineration, and the reuse and recycling of the plastic wastes have been used commonly to disposal of the wastes. Of these methods, recycling and reuse are preferable to reduce the amount of waste plastics. An attractive approach for the development of new composites, which combined wood-derived fillers with mixed waste plastics through melt-blending technique (Selke and Wichman 2004), has drawn the attention of the wood composites industry. Moreover, the use of wood-derived fillers for thermoplastics has been accepted by the plastics industry in recent years (Stark and Berger 1997). Compared to conventional inorganic fillers, wood-derived fillers have obvious advantages in acceptable specific strength and stiffness, lower density, lower volumetric cost, biodegradability (Karnani et al. 1997), and a natural renewable resource. Wood-derived fillers are least abrasive to the equipment during processing, which will reduce production cost considerably. However, the collected plastic wastes almost inevitably consist of several different kinds of plastics, and in practice, it is almost impossible to separate them completely. Therefore, the recycling and reuse of multicomponent polymer blends, which consist of at least three immiscible polymers, has the significance and attracts much attention (Ha et al. 1996; Hemmati et al. 2001). However, different kinds of the polymers are often immiscible, and this leads to poor compatibility between hydrophobic thermoplastic and hydrophilic wood filler. It is a real challenge to obtain an effective interfacial adhesion of multiphase system. Previous studies mainly focused on the improvement of the interactions between lignocellulosic fibers and polymer. Maleic anhydride grafted synthetic polymer has been

used as a compatibilizer, and proved to have a bridging effect between wood filler and the polymer matrix, resulting in improvement of material mechanical properties (Lu et al. 2000). Ha et al. (1999) reported that SEBS-MA worked well as a compatibilizer to enhance tensile properties and thermal stability in both virgin and recycled commingled blend systems. Joseph et al. (1996), Varghese et al. (1994) and Mokoena et al. (2004) studied the physical and tensile properties of sisal fiber/low-density polyethylene composites. It has been found that the addition of a small amount of peroxides leads to improvement in the tensile strength of the composite. In our previous works, the mechanical properties of wood fiber-PP/PE composites were improved through the grafting reaction of MAH onto PP/PE blends matrix (Gao et al. 2008).

In the present work, two modification methods were used to improve the mechanical properties of recycled plastic blends filled with wood flour by using SEBS-g-MAH compatibilizer and grafting maleic anhydride *in situ*. In addition, dynamic mechanical analysis (DMA) was used to evaluate the viscoelastic properties of wood flour/recycled plastic blends composites.

Material and methods

Materials

The plastic materials used in this study were virgin or recycled polypropylene, polystyrene and polyethylene. The virgin commercial polymers were obtained from Daqing Petroleum Company. They were high-density polyethylene (HDPE, 2200J, MFI: 6.0g/10min, ASTM D 1238), Polystyrene (PS, 200D, MFI: 8.0g/10min, ASTM D 1238) and Polypropylene (PP, T30S, homopolymer, MFI: 3.0g/10min, ASTM D 1238). The recycled PP and HDPE were obtained from Harbin Dongli Chemical Corporation as pellets. Recycled PS was prepared from waste foamed polystyrene by cutting, smashing and drying at 125°C into 1-2 mm diameter particles. Wood flour of *poplar* with average particle size of 250 μm (nominal 60 mesh), which was supplied by Harbin Yongxu Composites Co., Ltd, was dried at 105°C for 24 h. Maleic anhydride grafted styrene-ethylene- butylenes-styrene block copolymer (SEBS-g-MAH, GPM400C, total maleic anhydride/acid content of 1.5 wt%), was obtained from Ningbo Nengzhiguang New Materials Technology Co., Ltd. Maleic anhydride (MAH) was supplied by Tianjin Kermel Chemical Reagent Co., LTD, and dicumyl peroxide (DCP) was supplied by Shenyang Xinxi Reagent corporation.

Preparation of composites

Wood flour, virgin or recycled plastic mixture and additives were mixed in a high speed blender for 10 min, then extruded by a twin-screw/single-screw tandem extruder system with 11 heating zones, equipped with a 4 mm × 40 mm die and cooling accessory for composite strip preparation. All of the composites samples were prepared in the same processing conditions. The melt temperature was set below 190°C to avoid thermal degradation of the wood flour. The specimens for further study were directly cut from the extruded strips without undergoing any other hot pressing.

Mechanical tests

The mechanical tests were conducted according to GB/T standards for plastics. The mechanical properties were determined using a mechanical testing device (Shenzhen Reger Corporation, RGT-20A). Tensile test was performed according to GB/T 1040-1992 (ISO 527), with a crosshead speed of 5 mm/min; the gauge length was 50 mm. Flexural test was carried out according to GB/T 9341-2000 (ISO 178), with a testing speed of 2 mm/min, and the span was 60 mm. Charpy unnotched impact strength was measured with a XJ-50G impact tester according to GB/T1043-1993 (ISO 179). At least five replicates were used for each measurement.

Dynamic mechanical analysis (DMA)

Dynamic mechanical properties were determined on an AR2000ex rotational rheometry (TA Instruments) in the temperature range of −100°C to 170°C at a heating rate of 3°C/min, the delay time was 20 seconds. Liquid nitrogen was used to cool the samples. The 10-min temperature equilibrium time at the lowest temperature of −100°C was kept in order to sufficiently cool the specimens. The oscillating frequency was fixed at 1 Hz, and the constant strain amplitude of 0.05% was applied, which was well within the linear viscoelastic region. The specimen size is 55 mm long, 10 mm wide and 3 mm thick. Three specimens were made for each sample to check the reproducibility of results. The viscoelastic properties such as the storage modulus (G′) and loss factor (tanδ) of composites were obtained as a function of temperature from dynamic mechanical analysis.

Scanning electron microscopy (SEM) analysis

The breaking section of the composites was examined with an environment scanning electron microscope (FEI QUANTA200). Samples were fractured in liquid nitrogen and coated with gold in an ion-sputter coater before being examined with the microscope at an acceleration voltage of 20 KV.

Results and discussion

Mechanical properties

Table 1 summarized the mechanical properties of the virgin plastic blends with 30% and 40% wood flour. The results showed that the mechanical properties of the composites decreased with the increase of wood flour content from 30% to 40%. Especially, the impact properties decreased remarkably. In general, the increase of wood flour deteriorated the interfacial structure of the multiphase system and thus decreased the mechanical properties of the composite. The ratio of PP/HDPE/PS also significantly

influenced the mechanical properties of the composites. The major phase in the plastic blends played a dominant role in the properties of wood four filled plastic blends. The flexural and impact properties of 7/1.5/1/5 weight ratio of PP/HDPE/PS blend provided higher impact properties than other blends, regardless of whether it had 30% or 40% wood four, and the tensile strength was close to the highest value among the blends investigated. As a whole, the virgin plastic blends of PP/HDPE/PS (7/1.5/1.5) with 30% wood flour gave the better mechanical properties.

Table 2 showed the effect of DCP and MAH contents on mechanical properties of virgin plastic blends with 30% wood flour. The plastic blend composition was fixed at the weight ratio of 7/1.5/1.5 (PP/PE/PS). The results of mechanical properties demonstrated that the addition of DCP and MAH significantly improved the mechanical properties of the composites except the elongation at beak. The significant increase of tensile and flexural strength of the composites suggested that the interfacial adhesion between wood flour and plastic blends was improved. During the extrusion, DCP peroxide could initiate crosslinking or grafting reaction of MAH on the macromolecular chain *in situ*, resulting in an improvement in the compatibility between different plastics, and the MAH grafted plastic blends further reacted with wood flour by the esterification, forming the crosslink between plastic blends matrix and wood flour. Also, polar monomer MAH might be grafted to wood flour firstly by the esterification, and then crosslink with polymer matrix in the presence of DCP initiator, which facilitates the interact or improves the compatibility between polymer components and wood flour.

Table 1 Mechanical properties of virgin plastic blends with 30% and 40% wood flour

Sample Code	Polypropylene (%)	High-density polyethylene (%)	Polystyrene (%)	Wood Flour (%)	Tensile Strength (MPa)	Flexural Strength (MPa)	Flexural Modulus (GPa)	Charpy Impact Strength ($KJ·m^{-2}$)
1	49	10.5	10.5	30	19.7±0.3	37.2±1.6	2.1±0.4	7.2±0.9
2	10.5	49	105	30	17.5±0.4	30.2±1.3	2.0±0.2	4.5±0.5
3	10.5	10.5	49	30	20.8±0.9	36.2±1.3	2.7±0.1	3.1±0.2
4	42	9	9	40	15.1±1.5	32.3±2.4	2.1±0.3	4.3±0.6
5	9	42	9	40	14.3±1.7	30.3±0.6	2.0±0.1	3.8±0.1
6	9	9	42	40	13.8±1.9	24.9±1.2	1.9±0.1	2.5±0.2

The weight ratio of DCP/MAH maintained at 1/10, and the DCP content varied from 0.05 to 0.15 phr and MAH content from 0.5 to 1.5 phr. When 0.1 phr DCP and 1 phr MAH were added to the multiphase system, the tensile and flexural properties reached to their maximum levels. Further addition of DCP and MAH did not improve the strength and modulus, but caused a decrease in mechanical properties. It is possible that the loading of DCP higher than 0.1 phr would induce the degradation of polymer, and the loading of MAH higher than 1 phr might also produce some adverse reactions such as homopolymerization, resulting in the decrease of mechanical properties. However, if the loading of DCP was less than 0.1 phr and the loading of MAH was less than 1 phr, it would be difficult to initiate sufficient free radicals to graft or crosslink effectively in interfacial position of wood flour and polymer blends, consequently leading to a limited improvement in mechanical properties. Excess DCP would initiate further chain scission of the polymer matrix, and decrease the molecular weight of the polymer matrix.

Table 2 Mechanical properties of virgin plastic blends/wood flour composites with different dosage of dicumyl peroxide (DCP) and maleic anhydride (MAH)

(PP/PE/PS)/WF	DCP (phr)	MAH (phr)	Impact strength ($KJ·m^{-2}$)	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at Break (%)	Flexural strength (MPa)	Flexural modulus (GPa)
70(7/1.5/1.5)/30	0.00	0.00	7.2±0.9	19.7±0.3	2.2±0.3	2.7±0.3	37.2±1.6	2.1±0.4
70(7/1.5/1.5)/30	0.05	0.50	10.4±1.5	25.8±0.6	2.2±0.1	2.2±0.2	40.0±1.0	1.8±0.1
70(7/1.5/1.5)/30	0.10	1.00	9.5±1.0	27.7±1.8	2.3±0.1	2.1±0.2	44.1±2.4	2.2±0.2
70(7/1.5/1.5)/30	0.15	1.50	7.9±1.3	25.3±1.1	2.2±0.2	2.1±0.1	41.8±1.7	2.1±0.1

Fig. 1 (a) and (b) showed the effects of SEBS-g-MAH compatibilizer and the mixtures of MAH and DCP on the tensile strength of the virgin and recycled polymer blends with 30% wood flour. The addition of SEBS-g-MAH and the mixtures of DCP and MAH increased the tensile strength of virgin and recycled plastic blends composites. The composites with virgin plastic blends exhibited better mechanical properties than those of the composites with recycled polymer blends (as shown in Figs. 1 and 2), regardless of whether the system contained SEBS-g-MA or the mixtures of DCP and MAH. The inferior properties might result from the aging of the recycled polymer materials, the various uncertain additives, and contaminants in them, etc. The addition of DCP and MAH increased the tensile and flexural strength of composites to a higher level compared with SEBS-g-MAH (see Fig. 1 (a) and (b)). The results implied that the reactive compatibilization (adding DCP and MAH) was more effective to modify the blend systems *in situ* than an external compatiblizer (SEBS-g-MAH). The effect of SEBS-g-MAH

content on the tensile and flexural strength was shown in Fig. 1 (a) and (b). The SEBS-g-MAH content varied from 4 phr to 10 phr; the tensile and flexural strength showed a decreasing trend with increasing SEBS-g-MAH content although a little amount of SEBS-g-MAH (4 phr) has improved the strength of composites. This may be attributed to the fact that the maleic acid anhydride group in the backbone of the SEBS reacts with the hydroxyl groups at the wood flour surface (Hedenberg and Gatenholm 1995; Oksman et al. 1998).

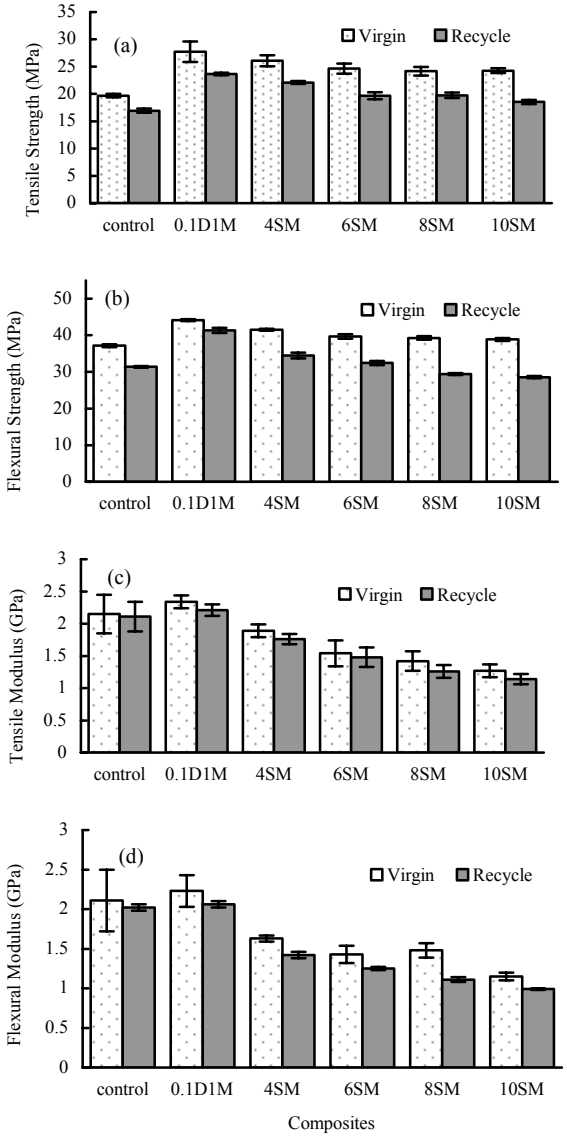

Fig. 1 Tensile strength (a), flexural strength (b), tensile modulus (c), and flexural modulus (d) of the composites of virgin and recycled plastic blends with 30% wood flour. Control, the composites without additives; 0.1D1M, blends with 0.1 phr DCP and 1 phr MAH; 4SM, blends with 4phr SEBS-g-MAH; 6SM, blends with 6 phr SEBS-g-MAH; 8SM, blends with 8 phr SEBS-g-MAH; 10SM, blends with 10 phr SEBS-g-MAH.

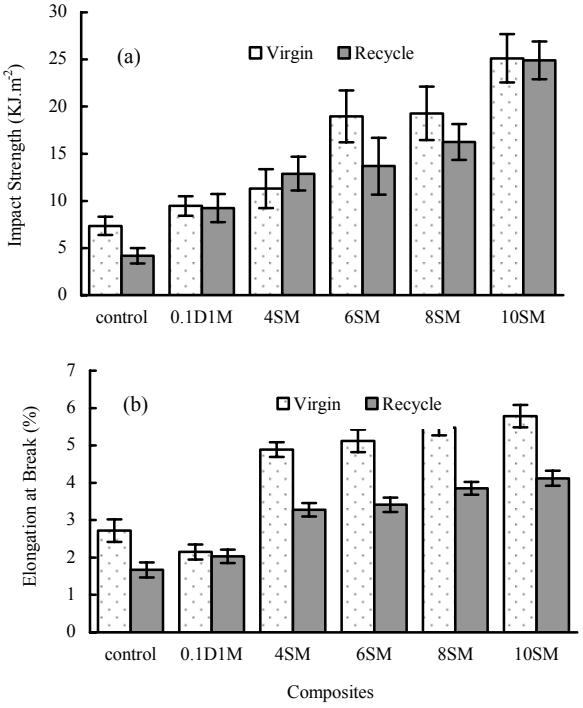

Fig. 2 Unnotched impact strength (a) and Elongation at break (b) of the composites of virgin and recycled plastic blends with 30% wood flour.

The effects of DCP/MAH and SEBS-g-MAH on the stiffness of the virgin and recycled polymer blends with 30% wood flour were shown in Fig. 1(c) and (d), respectively. The addition of 0.1 phr DCP and 1 phr MAH had a positive effect on increasing composites' stiffness, while, the addition of SEBS-g-MAH decreased the tensile modulus of composites possibly due to the low-modulus of the elastomer (Oksman and Clemons 1998). In this work, the tensile modulus of wood flour/virgin polymer blends composites decreased from about 2.15 GPa to about 1.27 GPa with the addition of 10 phr SEBS-g-MAH. The same trend was also observed for recycled polymer blends composites (from about 2.11 GPa to 1.14 GPa). Moreover, the addition of 10 phr SEBS-g-MAH into the recycled polymer blends composites decreased their flexural modulus.

The results of Charpy unnotched impact test were shown in Fig. 2(a). SEBS-g-MAH and the mixtures of DCP and MAH increased the impact strength. Especially, the content of SEBS-g-MAH significantly affected on the impact strength of virgin or recycled plastic blends with wood flour. The impact strength of wood flour/recycled plastic blends composites increased from 4.18 KJ/m^2, 12.89 KJ/m^2 and then to 24.91 KJ/m^2 when the loading of SEBS-g-MAH increased from 0 phr to 4 phr and then to 10 phr, respectively. The increase in unnotched impact strength was remarkable, this may be closely correlated with a soft elastomeric interphase formed around the wood flour, which can transfer stresses from the matrix to the wood flour and result in more energy demand to start crack propagation (Oksman and Lindberg 1998).

The elongation at break increased with the increase of SEBS-g-MAH content (Fig. 2(b)). As shown in (Fig. 2(b)), the 4 phr SEBS-g-MAH additions increased the elongation at break of recycled polymer blends composites by 96%, while the further increase was only about 25% with the increasing additions from 4 phr to 10 phr. However, the addition of DCP and MAH had a negative effect on elongation of break.

Dynamic mechanical properties

Dynamic mechanical analysis is an excellent tool to characterize the viscoelasticity of fiber reinforced thermoplastic material because the dynamical mechanical properties are highly sensitive to the structure of the composites (Tajvidi et al. 2003; Yang et al. 2007). Therefore, a better understanding of the dynamical mechanical properties will help to design and prepare the optimal composite.

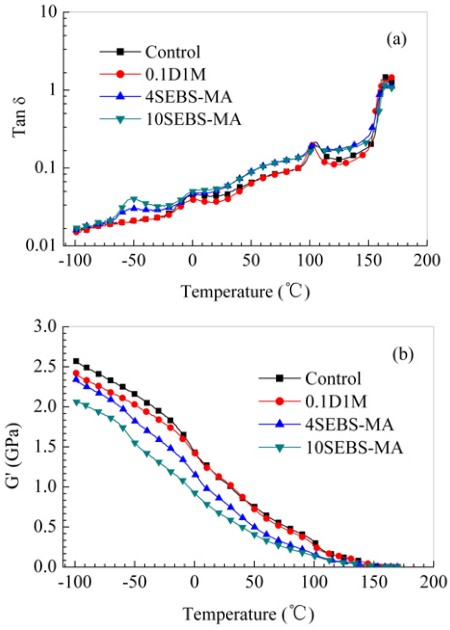

Fig. 3 Loss factor (tanδ) (a) and Storage modulus (G′) (b) of recycled plastic blends with 30% wood flour as a function of temperature.

The loss factor (tanδ) provides information on the relative contributions of the viscous and elastic components of a viscoelastic material (Yang et al. 2007). The variations of tanδ of composites with temperature were shown in Fig. 3(a). In the range of investigated temperature, two absent transition peaks were found in the control sample curve. The temperature at −1°C was considered the segmental motions in the non-crystalline phase of polypropylene matrix, and the second larger transition peak (104°C) was attributed to the polystyrene's glass transition temperature. The curve of 0.1D1M showed the similar shape feature compared to the control sample, but the second tanδ peak amplitude was reduced, indicating interfacial adhesion between wood flour and polymer blends was improved. However the PS tanδ peak position was shift to lower temperature slightly, implying the decrease of PS molecular weight. A new shift tanδ peak was found from the 4SEBS-MA or 10SEBS-MA curve at about −51.7°C. It was attributed to the ethylene/butylenes (EB) part of the SEBS-MA, which was especially in favor of improving impact properties of composites. SEBS-MA, as a grafted thermoplastic elastomer, has higher damping properties, so the tanδ curve of composites with the addition of SEBS-MA showed increasing damping amplitude. But, the decrease in tanδ peak value at 104°C suggest an improvement of interfacial compatibility between wood flour and PS. The molecular mechanism of the relaxation in composites is very complex and further work is necessary.

The storage modulus (G′) means the ability of a material to store deformational ability, which describes a material's elastic behavior, approximately similar to the Young or elastic modulus, or stiffness (Yang et al. 2007). Fig. 3(b) showed the temperature dependence of G′ for the control sample and modified composites. A general declining trend for all curves was observed when the composites were subjected to higher temperatures. The recycled polymer blends matrix was soften, and this led to the reduction of composites' G′ with increasing temperature. Below 0°C, the 0.1D1M sample showed a lower storage modulus compared with the control sample. It suggested that the molecular weight of recycled polystyrene plastic decreased with the addition of DCP and MAH. In Fig. 3(a), it was found that the second transition temperature (the glass transition temperature of PS) of the 0.1D1M sample was lower than that of the control sample. Perhaps the addition of DCP induced break of a small part of the molecular chain of PS. However, it is interesting that the two curves are coming to superposition at temperatures higher than 0°C approximately, which implied the interaction of macromolecule chain and wood flour did not decrease as compared with that of the control sample. The addition of SEBS-MA decreased the storage modulus of the composites, and further decreased when SEBS-MA loading increased from 4 phr to 10 phr. This phenomenon may be attributed to the lower elastic modulus of the thermoplastic elastomer itself.

Morphology of composite fracture

The SEM micrographs of the fractured surfaces of a recycled PP/HDPE/PS (7/1.5/1.5) blends containing 30% wood four with or without compatibilizer were shown in Fig. 4. The micrographs can provide a lot of valuable information about the morphology of recycled polymer blends/wood flour composites, and SEM observation is a best direct way to study the interfacial adhesion between the filler and matrix.

Fig. 4 (a) showed the morphology of a PP/HDPE/PS (7/1.5/1.5) blends with 30% wood flour without any addition of additives. The gross phase separation was observed. Fig. 4 (b) showed that the minor phase was pulled out, and holes were found. These observations indicate that the compatibility was very poor between polymer blends matrix. The microscopic information verified the poor mechanical properties of wood flour/recycled plastic blends composites. A gap between the wood particle and polymer matrix was observed in Fig. 4 (c), which indicates poor interaction between the wood surface and the polymer blends

matrix.

In Fig. 4 (d), it is difficult to distinguish the wood particles from the polymer matrix. The mechanical properties of the sample also showed the highest stiffness and strength, implying that the interfacial adhesion was enhanced between the wood four and the polymer blends matrix. Fig. 4 (e) revealed the finer morphology of the polymer matrix of the composites with addition of 10 phr SEBS-g-MAH. The interface of the dispersed phase and continuous phase was unobvious, and almost no voids were found. Fig. 4 (f) showed the microstructure of the wood flour/recycled polymer blends with 10 phr SEBS-g-MAH, showing a wood particle embedded in the polymer blends matrix.

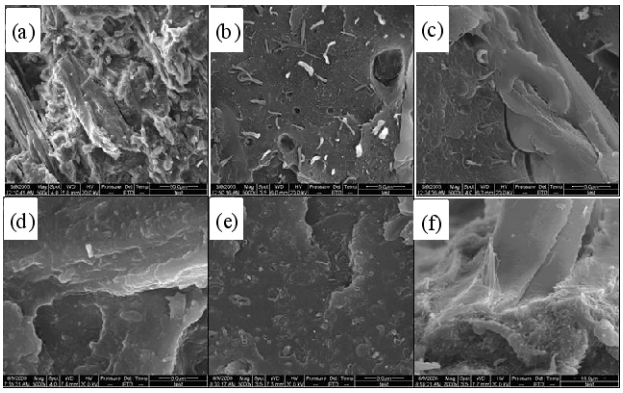

Fig. 4 SEM micrographs of wood flour(30%) and recycled polymer blends(PP/HDPE/PS: 7/1.5/1.5) composites with different treatment: (a), (b) and (c) without compatibilizer, (d) with addition of 0.1phr DCP and 1phr MAH, (e) and (f) with addition of 10phr SEBS-g-MAH.

Conclusions

Using the mixture of DCP and MAH as a reactive compatibilizer by extruding is an effective way to improve the mechanical properties of virgin and recycled plastic blends with wood flour except for impact strength and elongation at break. The addition of SEBS-g-MAH improved the tensile and flexural strength of composites, especially unnotched impact strength. However, the strength and stiffness of composites tend to decrease with increasing SEBS-g-MAH content. Comparing with SEBS-g-MAH, the mixtures of DCP and MAH resulted in a more significant increase in the strength and modulus though it caused a decrease in the elongation at break. SEBS-g-MA shows the potential to increase the impact strength of composites, but the mixtures of DCP and MAH can improve the interfacial adhesion of composites more than SEBS-g-MAH. Morphological study verified that the interfacial adhesion or the compatibility of polymer blends with wood flour was improved by adding SEBS-g-MAH or the mixtures of DCP and MAH. The dynamic mechanical analysis shows that the material damping properties, especially impact properties at lower temperature, were improved significantly, though the addition of SEBS-MA decreased the storage modulus of composites. The mixtures of DCP and MAH did not improve the storage modulus of composites and shifted the second tanδ peak position to lower temperature slightly.

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